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DOI: DOI: 10.31643/2023/6445.33
Metallurgy

Determination of the quality of special coke as a result of heat treatment of coal from the Shubarkol field

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Received: *November 12, 2022*
Peer-reviewed: *December 16, 2022*
Accepted: *January 27, 2023*

ABSTRACT

To date, there is a tendency to increase the pace of production in the field of ferrous metallurgy. The constant demand for steel products is accompanied by an increase in prices for raw materials, including carbon reducing agents. In the conditions of the domestic market of Kazakhstan, of great interest is the study and the possibility of using low-baking and non-baking coal as a raw material for the production of special coke used as a reducing agent in metallurgy, the relevance and expediency of which is also due to the resource conservation and energy efficiency program in the use of raw materials put forward by the Government of the Republic of Kazakhstan. In this article, as a result of the search for high-quality, alternative types of reducing agents used in the production of ferroalloys, experimental data of thermal oxidation treatment (coking) of long-flame, non-baking coals of the Shubarkol deposit (Kazakhstan) are presented. In laboratory conditions, during the experiments, the tested grade D coals with a fraction of 70-80 mm were subjected to temperature exposure at temperatures of 800, 850, 900, 950 °C with various preset heating speeds to determine the quality characteristics that meet the requirements for reducing agents for the metallurgical industry, in particular for the production of ferroalloys, in electro thermal, steelmaking, for agglomeration of iron and non-ferrous ores, etc. A technical analysis of long–flame coal was carried out, the volatile and moisture content of which are Vdaf – 44.5%, W – 14.8%, respectively. Also, the obtained special coke was evaluated by the content of volatile components as a result of heat treatment of coals from the Shubarkol deposit: the volatile content averaged 1.73-3.15%, the moisture was 0.73-1.65%.Based on the results of the studies, the possibility of obtaining a special coke from these types of coals with appropriate characteristics was shown.

Keywords: coals, reducing agent, special coke, coke, ferroalloy production, long-flame.

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Introduction

The use of new (alternative) carbon reducing agents from non–baking and low-baking coals for the production of ferroalloys is relevant today - this measure allows partially or completely eliminating the use of expensive traditional coke [[1], [2], [3], [4]].To use alternative reducing agents in the production of metals, it is necessary to meet the requirements of consumers in terms of technical and chemical compositions, electrical resistivity, reactivity, porosity, durability, etc. [[5], [6]].

The reduction of metals by carbon at high temperatures has been called carbon thermal reduction. Coke nut, anthracite, and special coke are used as carbon-containing reducing agents in the production of ferroalloys.

A feature of the carbon-thermal reduction of metals is the formation of carbides, which leads to the production of carbon-saturated alloys. Therefore, such reduction is used in cases where there are no restrictions on the content of carbon in the ferroalloy or its decarburization is carried out. Reduction of metals from oxides by carbon and

CO can be represented by the following reactions [1]:

$$Me_x O_y + yC = xMe + yCO \quad (1)$$

$$2Me_x O_y + yC = 2xMe + yCO_2 \quad (2)$$

$$Me_x O_y + yCO = xMe + yCO_2 \quad (3)$$

Thus, the reducing agent plays a crucial role in the production of ferroalloys.

The use of coal from the Shubarkol deposit for the production of coke is one of the examples of the use of these reducing agents in the production of ferroalloys [[7], [8], [9], [10], [11], [12]]. Coals are characterized by low ash content, as well as low sulfur and phosphorus content. The quality characteristics of this coal are presented in Table 1 [[13], [14], [15], [16]].

The special coke obtained from Shubarkol coal has a low ash content (8-10%), an average sulfur content (0.26%) and phosphorus (0.015%), also has a high reactivity for CO_2 (4.61 cm^3 / g·s), has a high resistivity (33.0 Ohm·cm), porosity is P = 62%, [[2], [3], [4], [5]].The technical characteristics of the special box are shown in Table 2 [[17], [18], [19]].

Table 1 – Characteristics of the quality of Shubarkol coal

Indicators	Unitsof measurement	Content
Workingmoisture– W^p	%	13.2
Analyticalmoisture– W^a	%	4.6
Yield of volatile substances of the analytical sample – V^a	%	39.8
The release of volatiles to a dry ash-free mass V^{daf}	%	44.1
Carboncontent– C^a	%	65.0
Ash content of the analytical sample – A^a	%	5.2
Sulfur– S	%	0.46
Lower heat of combustion Q^r_i	kJ/kg	23700

Table 2 – Technical characteristics of special coke

Material	Technicalcomposition,%		
	Ash	***Volatile***	***Moisture***
Special coke	8 – 10	15 – 25	12 – 15

The resulting special coke meets the basic requirements for carbon reducing agents for the ferroalloy industry and is successfully used at such enterprises as branches of TNC Kazchrome JSC Aktobe and Aksu ferroalloy plants.

Since this type of coal belongs to non-baking long-flame coals of the D brand, the production of special coke in conventional coke batteries is impossible, therefore, a thermal oxidation method of coking on grate grates is recommended [[18], [19], [20]].

Despite the large number of scientific works carried out in the field of production of special coke from non-baking coals of Kazakhstan, there is incomplete information about the features of the pyrolysis process of this type of coal and the dependence on the physical and physicochemical parameters of the special coke and on the parameters of coking [[17], [18], [19], [20], [21], [22], [23]].

For example, the increased humidity of the special coke has a negative effect, leading to a violation of the technological and electrical modes of the furnace by reducing the accuracy of the dosage of reducing agents. The high volatile content also has a negative impact on the technological process, in particular, leading to an increase in temperature and electrical conductivity, sintering of the charge and low fit of the electrodes. According to VUKhIN'sresearch, in order to produce a special coke for ferroalloy production, the volatile yield should be 8-9% to ensure the absence of resinous substances, which lead not only to sintering of charge materials, but to clogging of the reducing agent pores during pyrolysis, contributing to a decrease in the reaction surface and reactivity [4].

In turn, the stable content of humidity and volatile parameters contribute to an increase in the electrical resistance and reactivity of the special coke [[1], [5], [18], [19], [20]].

The experimental part

To assess the quality of the obtained special coke, laboratory experiments were carried out on thermo-oxidative coking of long-flame coal at various temperatures. Fractionated coals of the Shubarkol deposit were considered as the test material.

The purpose of this experiment was to study the quality of the special coke in terms of volatile content and humidity as a result of heat treatment.

To achieve this goal, the following tasks were defined:

- to carry out technical analysis of volatile substances and humidity of coal and special coke;
- to determine the change in the volatile content in long-flame coal during heat treatment at temperatures of 800, 850, 900 and 950 °C.

The main equipment used was a muffle electric furnace "SNOL", designed for analytical work with various materials and various types of heat treatment at temperatures up to 1300 °C. Temperature regulation and control is carried out by an electronic microprocessor-based thermostat working in conjunction with a thermocouple. The experimental program provided for heat treatment in accordance with a given heating of 800, 850, 900 and 950 °C with different heating speeds.

For the experiments, 12 groups of lump coal with a fraction of 70-80 mm were selected (Figure 1), 4 pieces of the starting material (three samples for each temperature level).

The choice of the 70-80 mm fraction was made on the basis of studies conducted on medium-temperature coking of long-flame coals [24].

The technical analysis carried out in accordance with GOST 10742-71 showed that the yield of volatile substances of the initial coal (V^{daf}) was 44.5%, humidity (W) – 14.8%.

Figure 1 – Coals of the Shubarkol deposit

In accordance with the specified heating, each group of fractional coal was placed in a furnace for heat treatment. Visual observation of changes in the shape of the sample was carried out at 300-500 °C and a given maximum temperature, as well as changes during cooling.

Cooling for each of the two groups was carried out to 600 °C and 400 °C, followed by immersion in water at 40 °C and natural cooling to room temperature for each third group. In the obtained samples, after cooling, the content of volatile substances and humidity were measured.

Figures 2-5 show the temperature dependences of the heat treatment of samples with specified heating modes up to temperatures of 800, 850, 900, 950 °C, to assess the behavior of samples under specified modes.

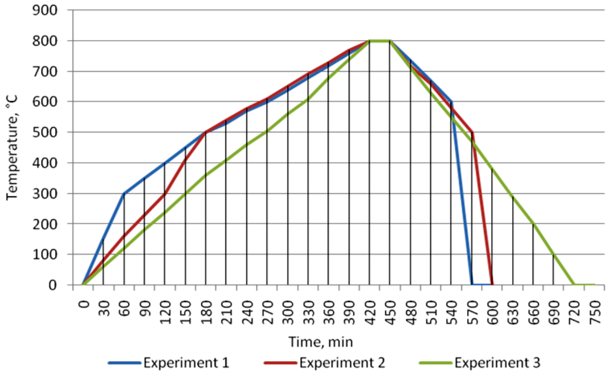


Figure 2 – Temperature dependence of heat treatment of samples up to 800 °C with 30 min exposure and cooling, experiments 1-3

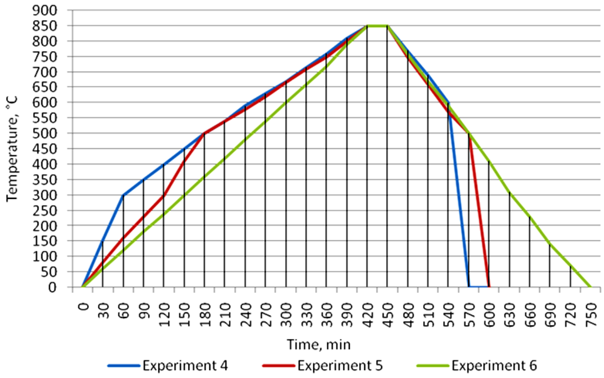


Figure 3 – Temperature dependence of thermal treatment of samples up to 850 °C with 30 min exposure and cooling, experiments 4-6

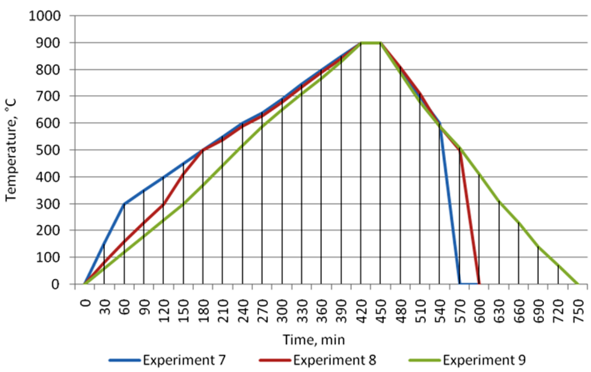


Figure 4 – Temperature dependence of heat treatment of samples up to 900 °C with 30 min exposure and cooling, experiments 7-9

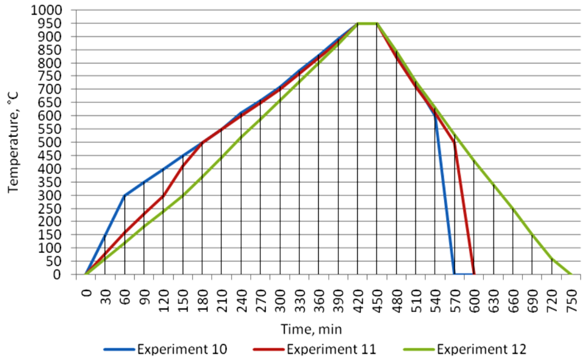


Figure 5 – Temperature dependence of heat treatment of samples up to 950 °C with 30 min exposure and cooling, experiments 10-12

According to the results of the data obtained during visual observation, heating of the samples to a temperature of 800 and 850 °C does not lead to a significant change in the shape of the samples and their destruction: there is a decrease in size, the formation of pores and microcracks.

At 900 and 950 °C, a slight decrease in indicators is observed: fractures and bevels are formed, a decrease in the size of samples with increased ash formation on the entire surface of the samples is noted.

Table 3 – Heating modes of the conducted experiments

Sample heating mode up to 800 °C		
Experiment1	***Experiment2***	***Experiment3***
heating up to 300 °C, speed 5 °C/min	heating up to 300 °C, speed 2.5 °C/min	heating up to 800 °C, speed 1.9 °C/min
heating from 300-500 °C, speed 4.2 °C/min	heating from 300-500 °C, speed 8.3 °C/min	
heating from 500 to 800 °C, speed 3.3 °C/min	heating from 500 to 800 °C, speed 3.3 °C/min	
cooling up to 600 °C, cooling in water at 40 °C	cooling up to 400 °C, cooling in water at 40 °C	naturalcooling
Sample heating mode up to 850 °C		
Experiment4	***Experiment5***	***Experiment6***
heating up to 300 °C, speed 5 °C/min	heating up to 300 °C, speed 2.5 °C/min	sample heating up to 850 °C at a heating rate of 2.02 °C/min
heating from 300-500 °C, speed 4.2 °C/min	heating from 300-500 °C, speed 8.3 °C/min	
heating from 500 to 800 °C, speed 3.3 °C/min	heating from 500 to 800 °C, speed 3.5 °C/min	
cooling up to 600 °C, cooling in water at 40 °C	cooling up to 400 °C, cooling in water at 40 °C	naturalcooling
Sample heating mode up to 900 °C		
Experiment7	***Experiment8***	***Experiment9***
heating up to 300 °C, speed 5 °C/min	heating up to 300 °C, speed 2.5 °C/min	sample heating up to 900 °C at a heating rate of 2.14 °C/min
heating up to 500 °C, speed 4.2 °C/min	heating up to 500 °C, speed of 8.3 °C/min	
heating from 500 to 900 °C, speed 3.75 °C/min	heating from 500 to 850 °C, speed of 3.75 °C/min	
cooling to 600 °C, further cooling in water at 40 °C	cooling to 400 °C, cooling in water at 40 °C	naturalcooling
Sample heating mode up to 950 °C		
Experiment10	***Experiment11***	***Experiment12***
heating up to 300 °C, speed 5 °C/min	heating up to 300 °C, speed 2.5 °C/min	sample heating up to 950 °C at a heating rate of 2.26 °C/min
heating up to 500 °C, speed 4.2 °C/min	heating up to 500 °C, speed 8.3 °C/min	
heating from 500 to 950 °C, speed 3.96 °C/min	heating from 500 to 950 °C, speed 3.95 °C/min	
cooling up to 600 °C, cooling in water at 40 °C	cooling to 400 °C, cooling in water at 40 °C	naturalcooling

Table 3 shows the values of the heating modes of the samples with the specified heating rates of all 12 experiments conducted.

Discussion of the results

1) Coking of coals to a temperature of 800 °C.

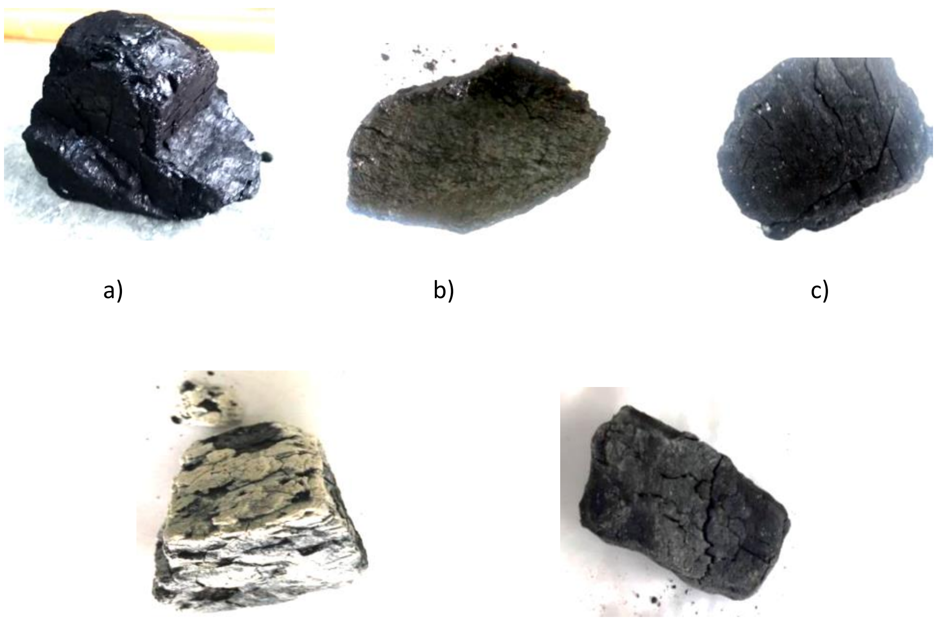
As a result of heat treatment, the obtained samples retained their original shape: up to a temperature of 300 °C, an endothermic reaction occurs with the absorption of heat and the formation of microcracks and micropores, then there is an abundant release of heat and gases, which affects the reduction in the size of coal, pores and cracks expand and increase. After slow cooling, there is a small ash content on the surface of the coke, which is washed off after cooling with water with the appearance of characteristic pigment spots in the places of salinity.

Figure 6 shows a photo image of the samples at different heating rates.

a) b) c) d)

a) heating up to 300 °C; b) heating up to 500°C; c) heating up to 800°C; d) cooling

Figure 6 – Coal samples as a result of the conducted experiment when heated to 800°C



a) b) c)

d) e)

a) heating up to 300 °C; b) heating up to 500 °C; c) heating up to 850 °C;
d) natural cooling to 400 °C; e) water cooling at 40 °C

Figure 7 – Coal samples as a result of experiments 4-6

2) Coking of coals to a temperature of 850 °C.

Heating up to 300 °C is accompanied by heat absorption with the formation of microcracks, the shape and characteristic luster of coal is preserved.

Further heating up to 600 °C is accompanied by gas release, an increase in the size and number of cracks and pores.

Further heating to 850 °C – the samples retained their shape, there is a slight decrease in size, and thereare no gas emissions. As a result of natural cooling, ash formation, microcracks, pores are observed on the surface; after cooling, characteristic spots and small bevels are present in the water at the sites of salinization.

The samples obtained are shown in Figure 7.

3) Coking of coals to a temperature of 900 °C.

Heating up to 300 °C is accompanied by the formation of small cracks, the samples have retained their shape, and there are no destructions, chips. Further heating up to 500 °C is accompanied by strong gas emission, an increase in the number of microcracks and pores.

When the temperature reaches 900 °C, the samples retained their shape, the surface is covered with deep microcracks, chips, and there are no gas emissions.

As a result of natural cooling, ash, microcracks, pores are present on the surface, there is an increase in bevels, samples have less strength; after cooling in water, and characteristic spots are present at the sites of salinization. The samples obtained are shown in Figure 8.

4) Coking of coals to a temperature of 950 °C.

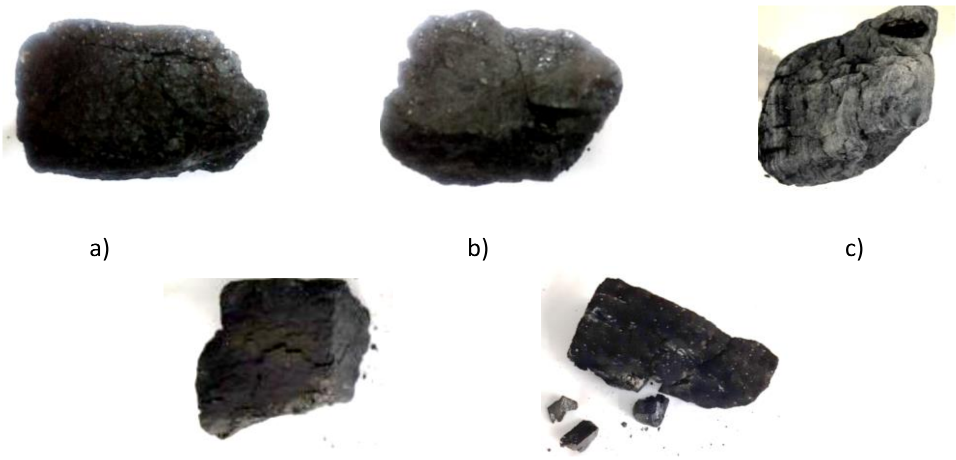
The samples obtained are shown in Figure 9.

Heating up to 300 °C is accompanied by the formation of small cracks, the samples have retained their shape, and there is no destruction, no chips. Further heating up to 500 °C is accompanied by strong gas emission, an increase in the number of microcracks and pores.

When the temperature reaches 950 °C, the destruction of samples is observed, the surface is covered with deep microcracks, chips, and there are no gas emissions.

As a result of natural cooling, ash, microcracks, pores are present on the surface; after cooling, characteristic spots are present in the water at the sites of salinization, the formation and increase in the size of bevels is observed, the destruction of samples is observed.

Data on the volatile content and humidity of the samples obtained from all the experiments are presented in Table 4.

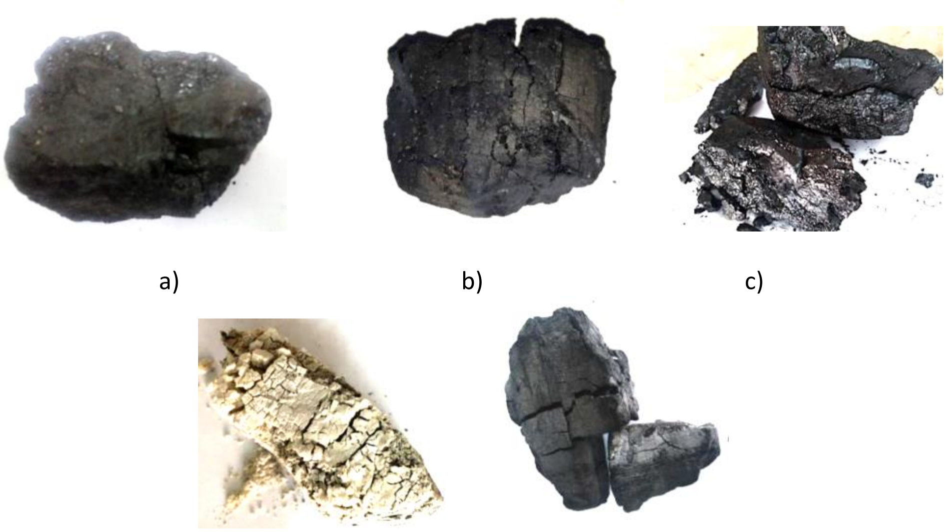


a) b) c)

d) e)

a) heating up to 300 °C; b) heating up to 500 °C; c) at 900°C; d) natural cooling; e) water cooling

Figure 8 – Coal samples as a result of the conducted experiment when heated to 900°C



a) b) c)

d) e)

a) heating up to 300 °C; b) at 400 °C; c) at 750 °C; d) natural cooling; e) water cooling

Figure 9 – Coal samples as a result of the conducted experiment

Table 4 – Data on volatile content and humidity

Core size class, mm	**Cooling after removal from the furnace at 600 °C.**		**Cooling after removal from the furnace at 400°C**		**Cooling to room temperature**	
	Experiment1		***Experiment2***		***Experiment3***	
	V	W	V	W	V	W
Inner core 0-5 mm	3.11	1.54	2.72	1.23	2.23	1.10
Inner core 5-10mm	3.19	1.39	2.54	1.88	2.30	1.18
Outer part 10-25 mm	3.04	1.26	2.45	1.91	3.03	1.20
Outer part 25-40mm	3.27	1.28	2.61	1.59	3.06	1.15
Average per piece	3.15	1.37	2.58	1.65	2.65	1.16
	Experiment4		***Experiment5***		***Experiment6***	
Inner core 0-5 mm	2.91	1.53	2.63	1.36	2.19	0.97
Inner core 5-10mm	2.97	1.40	2.51	1.48	2.27	1.01
Outer part 10-25 mm	2.99	1.28	2.42	1.61	2.61	1.12
Outer part 25-40mm	3.01	1.27	2.56	1.57	2.72	1.05
Average per piece	2.97	1.37	2.53	1.50	2.45	1.04
	Experiment7		***Experiment8***		***Experiment9***	
Inner core 0-5 mm	2.83	1.44	2.53	1.37	2.01	0.83
Inner core 5-10mm	2.85	1.49	2.43	1.45	1.97	0.87
Outer part 10-25 mm	2.73	1.41	2.37	1.53	1.85	0.91
Outer part 25-40mm	2.91	1.47	2.44	1.49	1.73	0.99
Average per piece	2.83	1.45	2.44	1.46	1.89	0.90
	Experiment10		***Experiment11***		***Experiment12***	
Inner core 0-5 mm	2.21	1.42	1.97	1.40	1.85	0.75
Inner core 5-10mm	2.15	1.44	2.01	1.45	1.79	0.78
Outer part 10-25 mm	2.30	1.38	1.91	1.52	1.65	0.73
Outer part 25-40mm	2.31	1.41	1.85	1.51	1.61	0.81
Average per piece	2.24	1.41	1.93	1.47	1.73	0.77

According to Table 4, as a result of thermal exposure to the coals of the Shubarkol deposit, the samples have the required humidity and volatile values required for reducing agents.

In experiments 6, 9, 12, a decrease in humidity indicators is observed due to the high rate of coking.

At the same time, when temperatures reach 900 and 950 °C, there is a decrease in the size of the pieces and their integrity, the formation of bevels and, according to visual signs, strength.

Conclusions

In this work, experiments were carried out to assess the quality of non-baking coals of the Shubarkol deposit, and the influence of temperature conditions on the quality of samples of special coke obtained in laboratory conditions, used as reducing agents for metallurgical production, was investigated.

In total, 12 experiments of thermal exposure to long-flame coals of the Shubarkol deposit were carried out in the temperature range up to 800...950 °C with different preset heating speeds.

s a result, the following results were obtained:

- technical analysis of long–flame coal with a grain size of 70-80 mm, volatile V^{daf} – 44.5%, humidity W – 14.8 %;
- samples were obtained as a result of heat treatment up to 800, 850, 900 and 950 °C of long-flame coals with a size of 70-80 mm;
- the assessment of the special coke on the content of volatile components as a result of heat treatment of coals of the Shubarkol deposit was carried out: the content of volatile on average is 1.73-3.15%, the humidity was 0.73-1.65%.

Based on the obtained research data, the following coking mode is recommended:

- coal size 70-80 mm;
- coking temperature800-850 °C;

The evaluation of the obtained results proves the effectiveness of using long-flame coal from the Shubarkol deposit as reducing agents for the metallurgical industry, in particular ferroalloy production, electrothermal, steelmaking productions, for agglomeration of iron and non-ferrous ores and other.

Conflict of interest. On behalf of all the authors, the correspondent author declares that there is no conflict of interest.

Cite this article as: Aubakirov AM, Kaliakparov AG, Tolymbekova LB. Determination of the quality of special coke as a result of heat treatment of coal from the Shubarkolskoye field. Kompleksnoe Ispolzovanie Mineralnogo Syra = Complex Use of Mineral Resources. 2023; 326(3):96-106. https://doi.org/10.31643/2023/6445.33

Шұбаркөл кен орнының көмірін термиялық өңдеу нәтижесінде арнайы кокстың сапасын анықтау

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Мақала келді: *12 қараша 2022*
Сараптамадан өтті: *16 желтоқсан 2022*
Қабылданды: *27 қаңтар 2023*

ТҮЙІНДЕМЕ
Бүгінгі таңда қара металлургия саласындағы өндіріс қарқынын арттыру үрдісі байқалады. Болат өнімдеріне тұрақты қажеттілік шикізат бағасының, оның ішінде көміртекті тотықсыздандырғыштарға өсуімен қатар жүруде. Қазақстанның ішкі нарығы жағдайында металлургияда тотықсыздандырғыш ретінде қолданылатын арнайы кокс алу үшін шикізат ретінде төмен жентектелген және жентектелмейтін көмірлерді пайдалану мүмкіндігін зерттеу үлкен қызығушылық тудырады, оның өзектілігі мен мақсаттылығы ҚР Үкіметі ұсынған шикізатты пайдаланудағы ресурстарды үнемдеу және энергия тиімділігін арттыру бағдарламасына да байланысты. Бұл мақалада ферроқорытпалар өндірісінде қолданылатын тотықсыздандырғыштардың сапалы, балама түрлерін іздеу нәтижесінде Шұбаркөл кен орнының (Қазақстан) ұзын жалынды, күйежентектелмейтін көмірді термо тотықтырғыш өңдеудің (кокстеудің) тәжірибелік деректері келтірілген. Зертханалық жағдайда, тәжірибелер

жүргізу барысында 70-80 мм фракциясы бар Д маркалы сыналатын көмірлер сапалық сипаттамаларын анықтау үшін әртүрлі берілген қыздыру жылдамдықтарымен 800, 850, 900, 950 °C температурада температуралық әсерге ұшырады металлургия өнеркәсібі үшін, атап айтқанда ферроқорытпалар өндірісі үшін, электротермиялық, болат балқыту өндірісінде, темір және түсті кендерді агломерациялау үшін және т. б. тотықсыздандырғыштарға қойылатын талаптарға сәйкес келеді. Ұзын жалынды тас көмірге техникалық талдау жүргізілді, олардың ұшпа және ылғал мөлшерлері, сәйкесінше Vdaf – 44,5%, W – 14,8% құрайды. Сондай-ақ, Шұбаркөл кен орнының көмірін термиялық өңдеу нәтижесінде ұшпа компоненттердің құрамы бойынша алынған арнайы коксты бағалау жүргізілді: ұшқыштардың құрамы орта есеппен 1,73-3,15%, ылғалдылығы 0,73-1,65% құрады. Жүргізілген зерттеулердің нәтижелері негізінде тиісті сипаттамалары бар көмірдің аталған түрлерінен арнайы кокс алу мүмкіндігі көрсетілді.

Түйін сөздер:көмір, көмір, тотықсыздандырғыш, арнайы кокс, кокс, ферроқорытпа өндірісі, ұзын жалынды.

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Опытное определение качества спецкокса в результате термической обработки углей Шубаркольского месторождения

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Поступила: *12 ноября 2022*
Рецензирование: *16 декабря 2022*
Принята в печать: *27 января 2023*

АННОТАЦИЯ

На сегодняшний день наблюдается тенденция наращивания темпа производства в области черной металлургии. Постоянная потребность на стальную продукцию сопровождается с ростом цен на сырье, в том числе и на углеродистые восстановители. В условиях внутреннего рынка Казахстана большой интерес представляет изучение и возможность использования слабоспекающихся и неспекающихся углей в качестве сырьевого материала для производства спецкокса, использующегося в качестве восстановителя в металлургии, актуальность и целесообразность которого также обусловлена в рамках программы ресурсосбережения и энергоэффективности в использовании сырьевых ресурсов, выдвигаемых Правительством РК. В данной статье, в результате поиска качественных, альтернативных видов восстановителей, применяющихся в производстве ферросплавов, представлены экспериментальные данныетермоокислительной обработки (коксования) длиннопламенных, неспекающихся углей Шубаркольского месторождения (Казахстан). В лабораторных условиях, в процессе проведения опытов, испытуемые угли марки Д фракцией 70-80 мм подвергались температурному воздействию при температурах 800, 850, 900, 950 °C с различными заданными скоростями нагрева для определения качественных характеристик, соответствующих требованиям к восстановителям для металлургической промышленности, в частности для производства ферросплавов, в электротермическом, сталеплавильном производстве, для агломерации железных и цветных руд и т.д. Был проведен технический анализ каменного длиннопламенного угля, содержание летучих и влаги которых составляют, соответственно, V_{daf} – 44,5 %, W – 14,8 %. Также, проведена оценка полученного спецкокса по содержанию летучих компонентов в результате термической обработки углей Шубаркольского месторождения: содержание летучих в среднем составляет 1,73-3,15 %, влажность составила 0,73-1,65%. На основании результатов проведенных исследований, была показана возможность получения спецкокса из данных типов углей с соответствующими характеристиками.

Ключевые слова: угли, восстановитель, спецкокс, кокс, производство ферросплавов, длиннопламенный.

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